This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No 730871.

WP15 - Thin Film for Superconducting RF (TF-SRF)

Oleg Malyshev
WP15 coordinator
on behalf of the team

3rd ARIES Annual Meeting , 22 April 2020

WP Description

- The aim of this work package is to intensify systematic studies and development of the coating technology of superconducting materials to enable the superconducting coated RF cavities with $Q(E)$ characteristics better than for the bulk ones.
- The main emphasis will be on a systematic study of correlation between
  - surface preparation,
  - deposition parameters,
  - film structure, morphology, chemistry
  - as well as AC and DC superconductivity parameters
    - such as T_c, H_c, H_{fp}, H_{sh}, RRR
    - of superconducting material Nb, NbN, Nb_3Sn, MgB_2, etc.
    - deposited on Cu and bulk Nb,
  - and, finally, the behaviour at radiofrequency with the test cavities recently built at CERN, HZB and STFC.

What should be achieved

The main emphasis is on a systematic study of correlation between

TASK 15.2

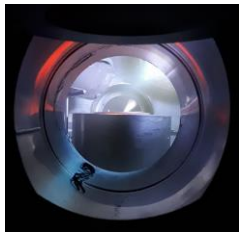
TASK 15.3

TASK 15.4

Surface preparation
- Cleaning, etching,
- Polishing, passivating

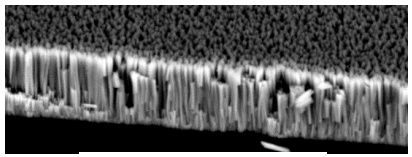
Thin film deposition
- **PVD**: DC, pulsed, HIPIMS…
- (PE)CVD, (PE)ALD
- **Nb, NbN, Nb_3Sn**, MgB_2, etc.

Film deposition



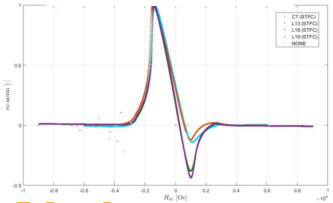
Film characterisation
- SEM, FIB, AFM,
- XPS, XRD, RBS, TEM…

SEM image of a NbN thin film

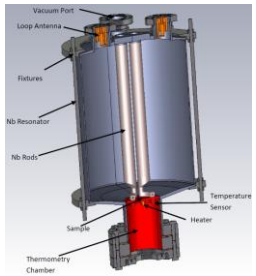


Superconducting properties measurement
- *RRR*, H_c, H_{fp}, H_{sh}, …
- AC and DC magnetic susceptibility,
- Field penetration

DC magnetisation



QPR



Superconducting RF properties evaluation
- QPR at CERN
- QPR at HZF
- HW cavity at ASTeC

Real cavity measurement
- Cavity deposition

WP15 Partners

	Participants	Leading	Participating
1	CEA (Saclay, France)		Task **4**
2	*CERN (Geneva, Switzerland)*		All tasks
3	IEE-SAS (Bratislava, Slovakia)		Tasks **4**
4	LNL/INFN (Legnaro, Italy)	Task 2	Tasks 1, **2** and **3**
5	Helmholtz-Zentrum Berlin (Berlin, Germany)	Task 4	Tasks 1 and **4**
6	RTU (Riga, Latvia)		Task 2 and **3**
7	University Siegen, (Siegen, Germany)		Tasks **3**
8	ASTeC/STFC (Daresbury, UK)	WP and Tasks 3	All tasks
9	Lancaster University (Lancaster, UK)		Task **4**

3rd year objectives

- Work in a collaboration following an agreed plan:
  - **Thin film development** on small samples (53 mm × 53 mm)
    - Nb, NbN, Nb_3Sn and SIS deposition (STFC, Siegen)
      - Laser treatment of the film (RTU)
    - Film characterisation (STFC, Siegen)
    - DC and AC superconductivity evaluation measurements (IEE, STFC))
  - **QPR samples for SRF test**
    - Sample manufacturing (HZB)
    - Cases for sample transfer (HZB)
    - Sample polishing with EP and SUBU (INFN)
    - Nb film deposition (STFC, Siegen)
    - Testing QPR (HZB, CERN)
- Meeting milestones and deliverables, writing reports

Task 15.3

Task 15.4

Task 15.4

Task 15.2

Task 15.3

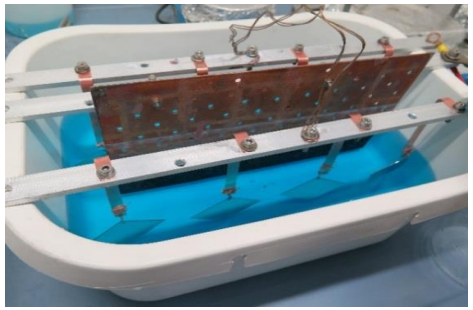
Task 15.4

This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No 730871.

Thin film development

Task 15.2. Substrate surface preparation

- 50 planar copper samples with 4 different procedures:
  - 50 samples with a size of 53mm x 53 mm were cut at CERN from the same copper sheet
    - 25 samples were treated at CERN with
      - SUBU solution
    - 25 samples were treated at INFN with
      - SUBU solution,
      - Electropolishing (EP),
      - SUBU+EP,
      - Tumbling



SUBU and Electropolishing treatments
Courtesy of E. Chyhyrynets and C. Pira (INFN)

- Based on results from 1st and 2nd year EP and SUBU were selected as most promising polishing procedures for future WP15 work
- This year work:
  - Samples were deposited at Siegen and STFC (Task 15.3)
  - Film characterisation in 4 institutes: INFN, RTU, Siegen and STFC
  - Superconductivity properties studied in at IEE (Task 15.4)

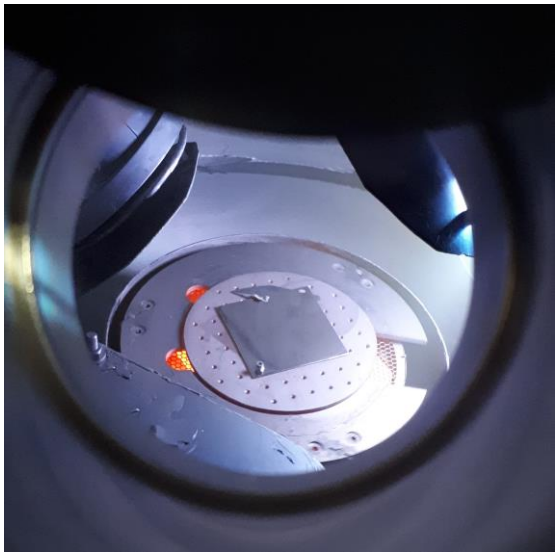
Task 15.3. Thin film deposition

- Deposition at facilities at different institutes:

University Siegen

A sample and a Nb target in deposition facility at University Siegen.
Courtesy of M. Vogel (Siegen)

ASTeC/STFC



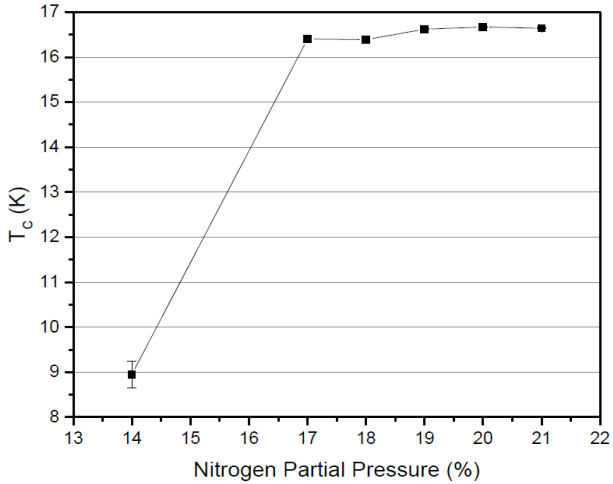
A sample during the Nb deposition at ASTeC/STFC

- Key facilities for the project – **deposition of the sample films**
- Quality of Nb films deposited in year 1 and 2 at INFN, Siegen and STFC is comparable
- This year the main focus is on producing and testing films different from Nb

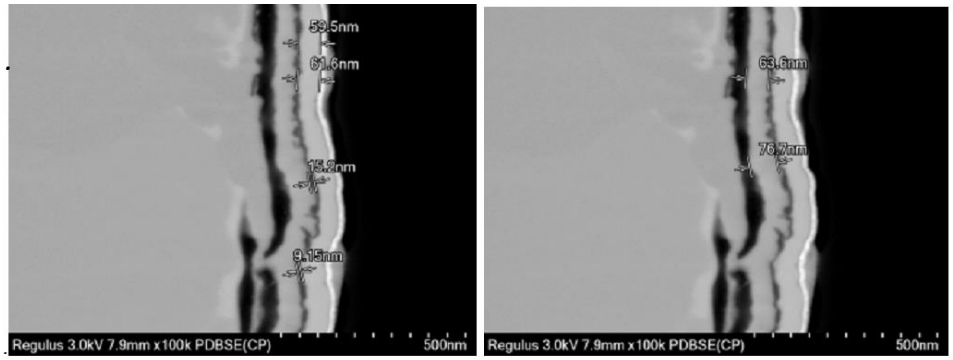
Thin film characterisation at STFC

- Samples deposited on the electropolished Cu substrates:
  - System 1 (Nb_3Sn) samples
  - System 3 (NbTiN) samples
  - Nb/Nb_3Sn
  - $Nb/AlN/Nb_3Sn$ (SIS) on Cu, Ta and sapphire

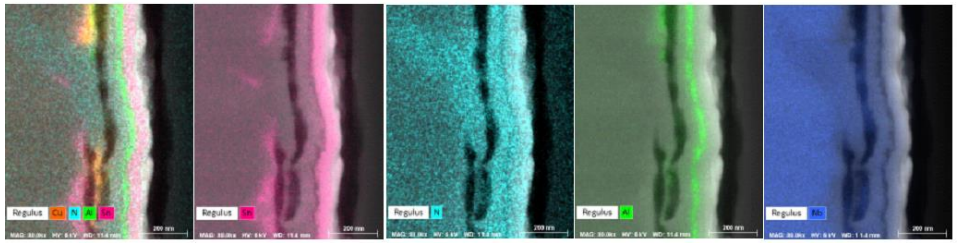
T_c as a function of (a) N_2 % for NbTiN samples



(a)



(b)



(a) High resolution SEM of ion milled X-section of SIS multilayer structure (Nb/AlN/Nb3Sn) deposited on Ta.

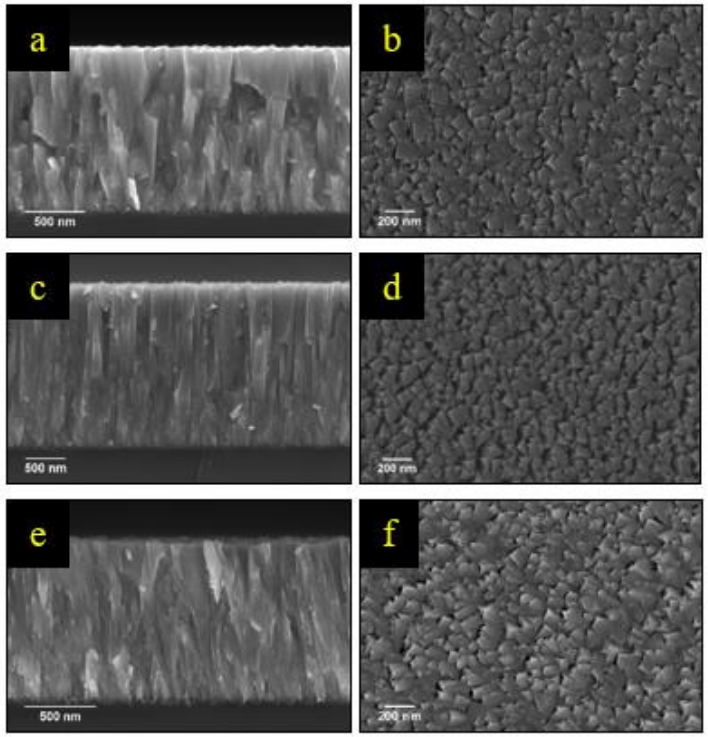
(b) EDX chemical mapping of the X-section.

Courtesy of R. Valizadeh (STFC)

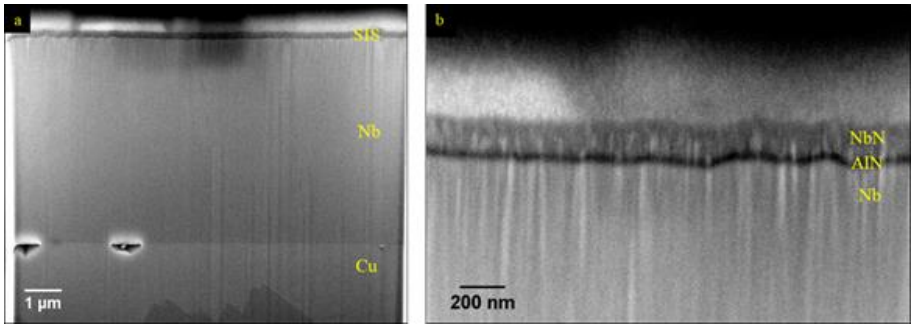
Thin film characterisation at Siegen

- Samples deposited on the electropolished Cu substrates:
  - 30 System 2 (NbN) samples
  - 4 Superconductor-Insulator-Superconductor (SIS) coatings

SEM images of optimised samples

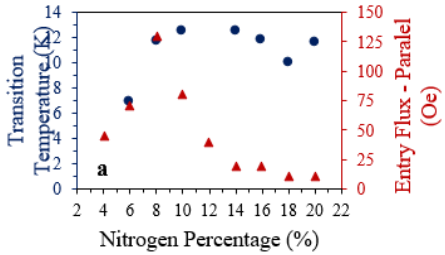


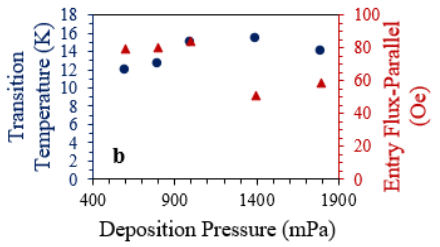
Cross sectional SEM images of Nb/AlN/NbN



Courtesy of M. Vogel (Siegen)

T_c and B_{en} in parallel field as a function of (a) N_2 %, (b) deposition pressure

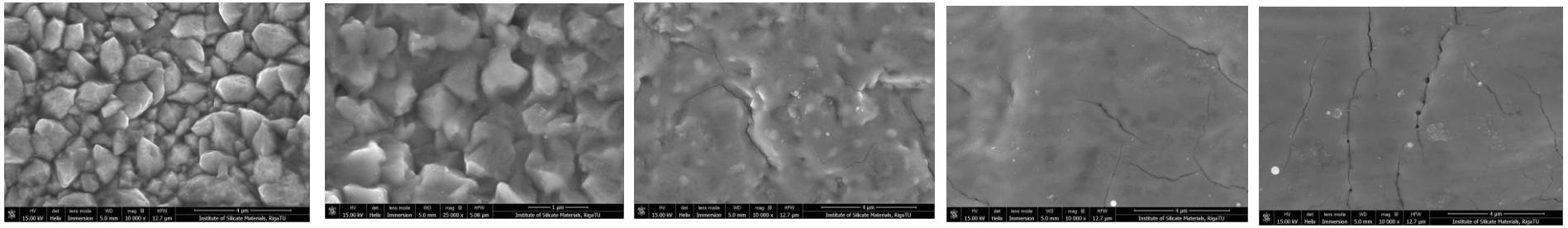




Post deposition laser treatment at RTU

- Aims:
  - Increase the grain size of Nb;
  - Increase the adhesion of Nb layer to Cu substrate (Annealing the defects by laser radiation).

a) b) c) d) e)



SEM images of Nb/Cu structure before irradiation (a) and after irradiation by Nd:YAG laser with I_1 = 140 MW/cm^2 (b); I_2 = 170 MW/cm^2 (c); I_3 = 253 MW/cm^2 (d); I_4 = 320 MW/cm^2 (e).

Courtesy to Arturs Medvids, Pavels Onufrijevs and Jevgenijs Kaupuzs (RTU)

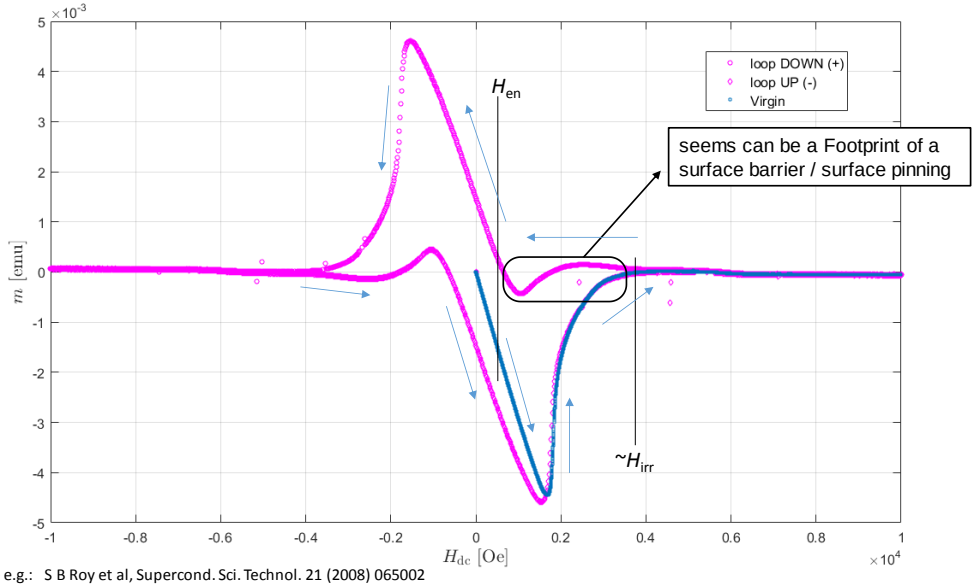
Preliminary results:

- The sizes of Nb crystals can be increased by laser radiation
- Defects between grains (pinholes) can be eliminated by laser radiation.

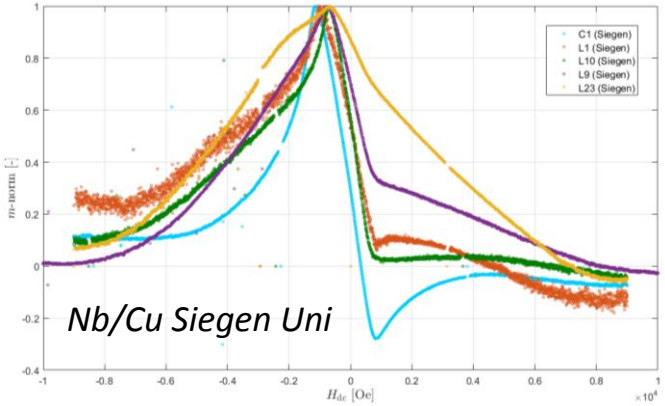
DC Superconductivity evaluation at IEE

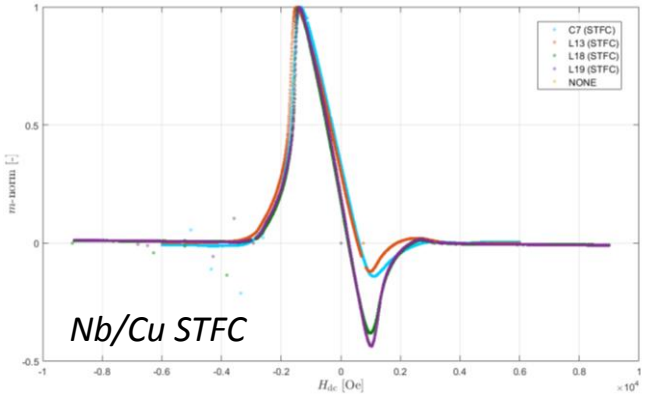
- PPMS (Physical Property Measurement System)
  - Virgin DC magnetisation curve: B_{en}(~$B_{c1\ perp}$), [B_p, B_{c2}]

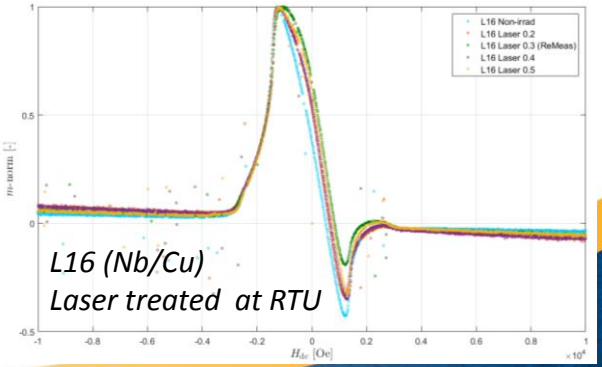
Shape of magnetization loop



see e.g.: S B Roy et al, Supercond. Sci. Technol. 21 (2008) 065002
E H Brandt, Physica C 332 (2000) 99–107
A S Dhavale et al, Supercond. Sci. Technol. 25 (2012) 065014

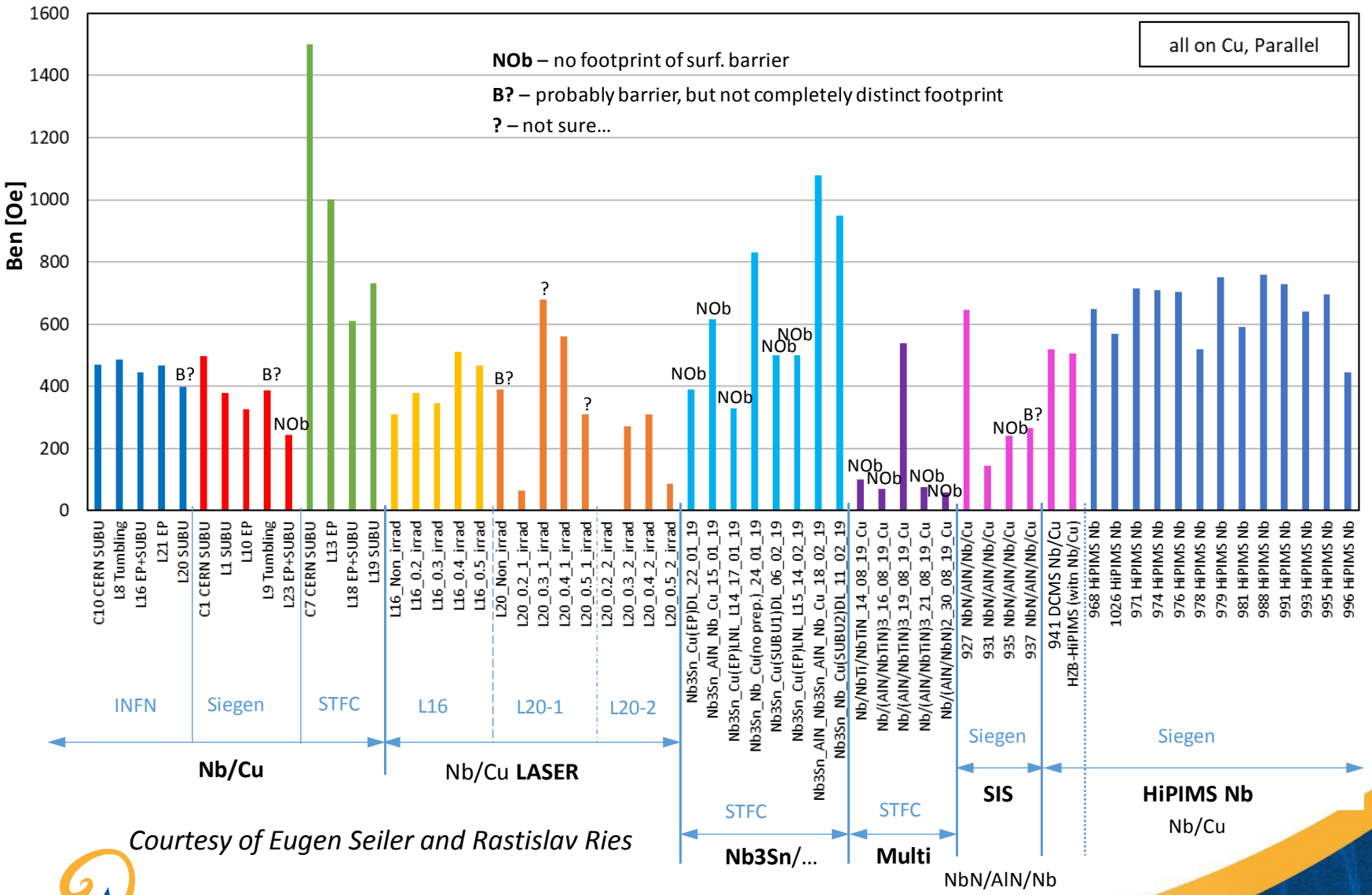






Courtesy of Eugen Seiler and Rastislav Ries (IEE)

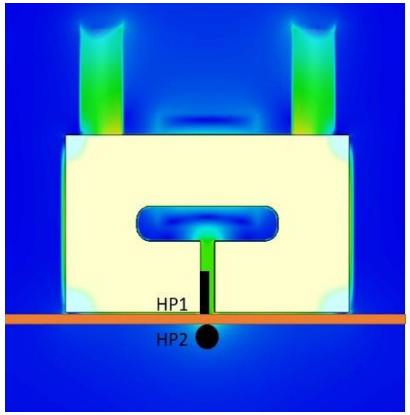
DC Superconductivity evaluation at IEE



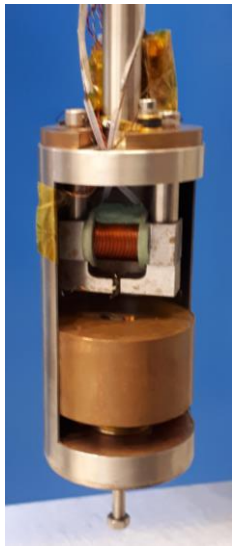
Courtesy of Eugen Seiler and Rastislav Ries

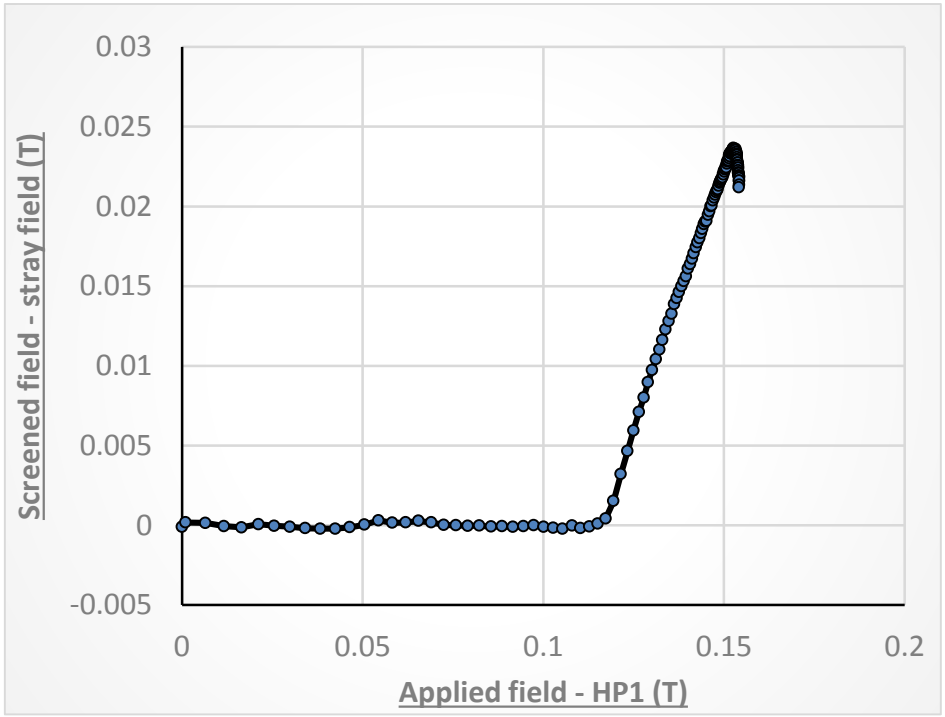
Task 15.4: AC/DC Superconductivity evaluation at STFC

- First test of the principle for magnetic field penetration for the planar sample
  - at LHe cryostst – **it works!**
  - but LHe gets more and more expensive



Courtesy of D. Turner (LancU/STFC)





- New dry facility has been designed and assembled.
- Testing and operation – after COVID-19 quarantine

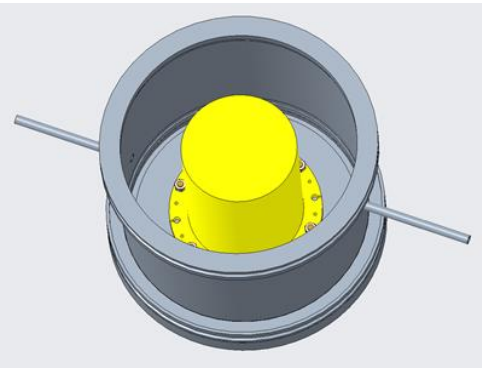
This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No 730871.

QPR samples for SRF test

QPR samples

Cu-Nb samples at HZB after production

Nb samples at HZB after production
Courtesy of Oliver Kugeler (HZB)

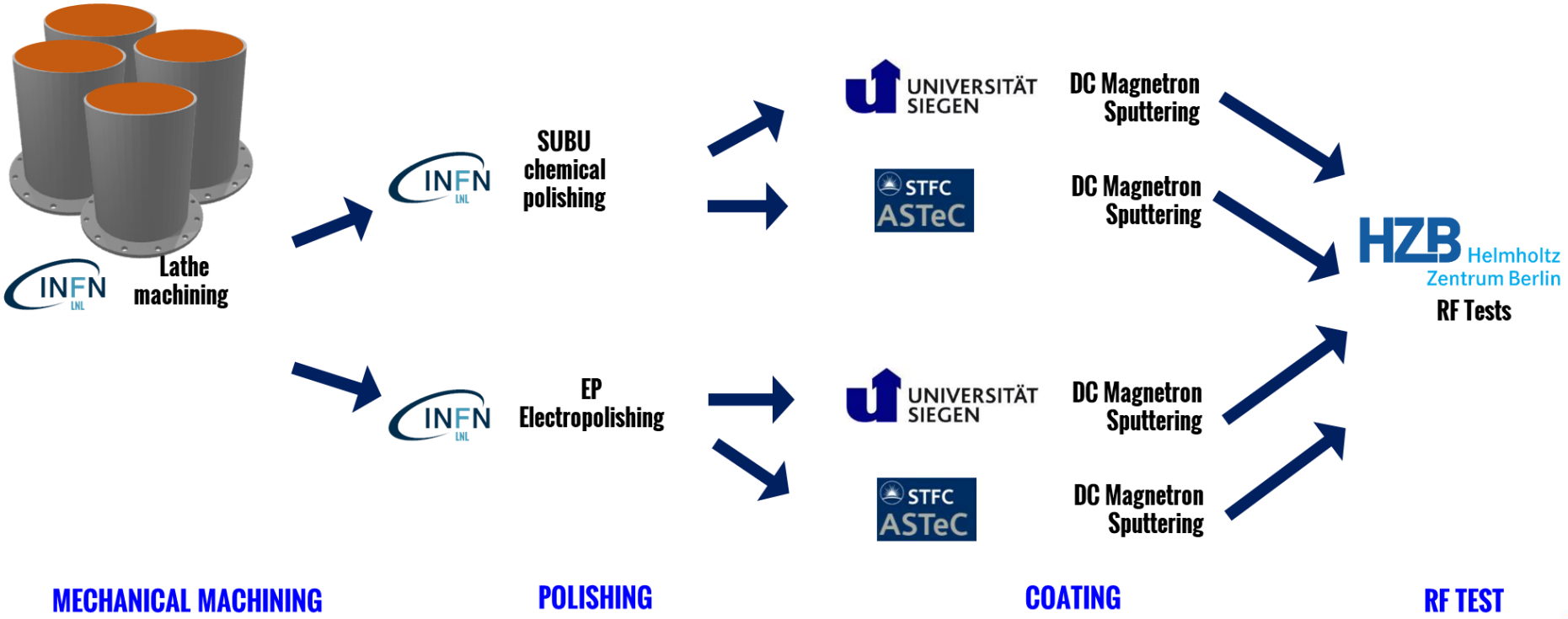


Sample transport chamber

- 5 OFHC samples, 5 Nb samples for QPR were ordered at Research Instruments and delivered in 2018
- A dedicated chamber for transporting the QPR samples under clean room conditions in vacuum or arbitrary atmospheres has been designed and manufactured:
  - It consists of ISO-KF160 standard pieces,
  - equipped with suitable fixtures for the samples,
  - and an evacuation manifold.

Workflow of the QPR Experiment

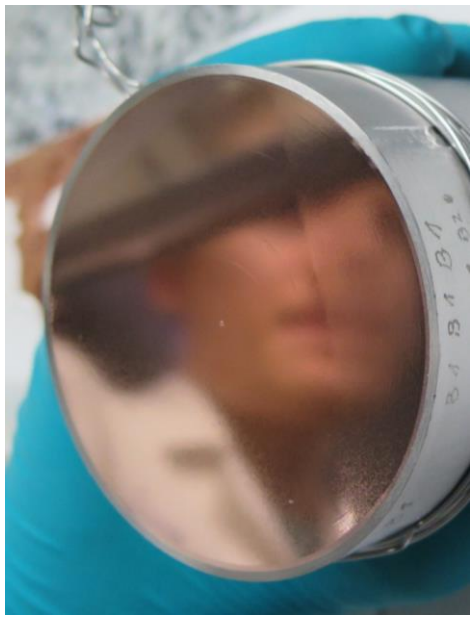
GOAL: Evaluate the effect of planar substrate Cu polishing on RF performance of QPR

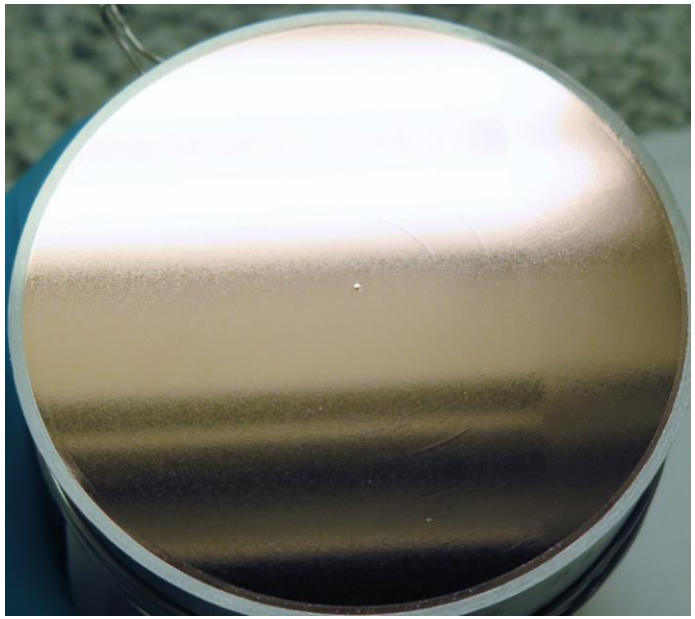


Courtesy of E. Chyhyrynets and C. Pira (INFN)

QPR substrate polishing at INFN

- SUBU5 (Chemical Polishing) -> 3 samples:
  - Sulfamic acid – 5 g/l, $(NH_4)_3Cit$ – 1 g/l, H_2O_2 – 50 ml/l, Butanol – 50 ml/l,
  - T = 73 °C
  - Average removal thickens: δ = ~6 μm
- Passivation (5 min):
  - Sulfamic acid – 20 g/l,
- EP (ElectroPolishing):
  - *Phosphoric acid 85% - 3 v.r., N-Butanol 98% - 2 v.r.,*
  - Temperature 40 °C
  - Average removal thickens: δ = ~15 μm
- Passivation:
  - Sulfamic acid – 20 g/l,



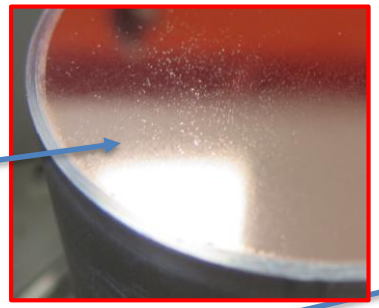
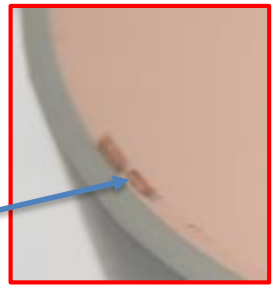


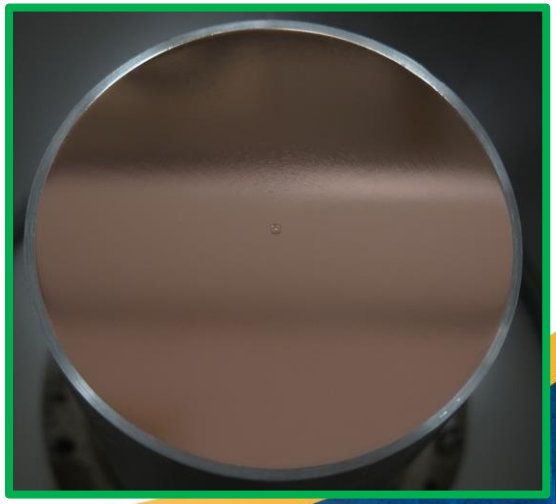
Courtesy of Eduard Chyhyrynets (INFN)

QPR substrate polishing – resolving problems

- Lathe machining
  - Middle point
  - Partial machining
  - Pitting
  - Nb rough edges
- SUBU5 polishing
  - Pitting
  - Stains and oxidations
  - Brazing material oxidation
- EP polishing
  - Pitting
  - Removal speed
  - Oxidations of Nb
- Solutions found for each problem



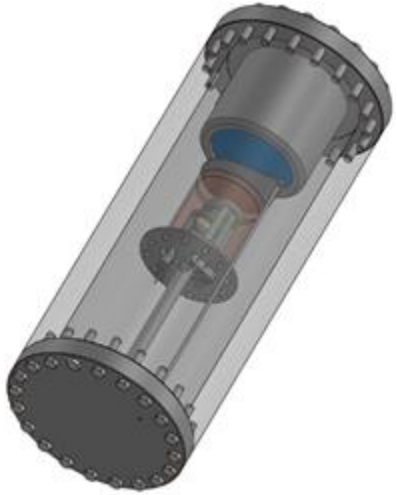





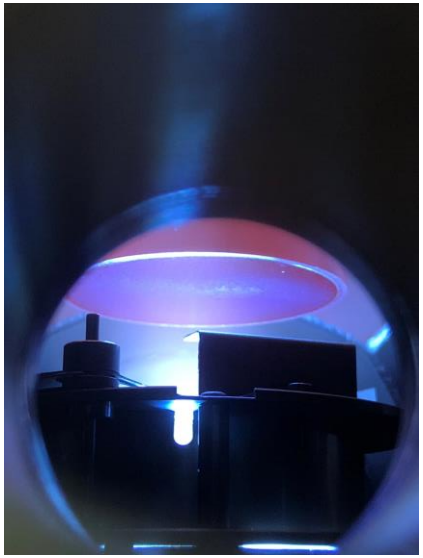
Courtesy of E. Chyhyrynets and C. Pira (INFN)

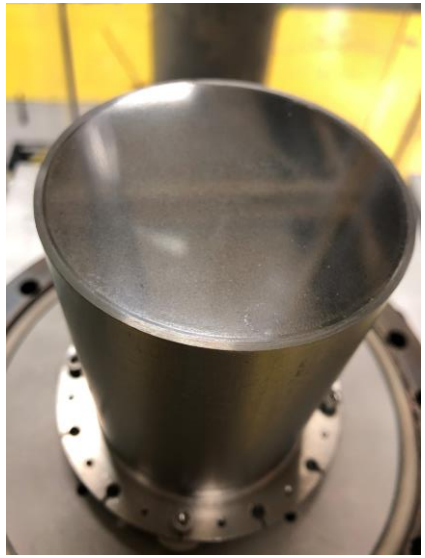
QPR deposition

A QPR sample inside the deposition chamber at Siegen Uni. Courtesy of M. Vogel



Design of new QPR sample holder for deposition at INFN. Courtesy of C. Pira





A QPR sample (a) inside the deposition chamber and (b) after Nb film deposition at STFC. Courtesy of R. Valizadeh (STFC)

Five QPR samples has been deposited with Nb at Siegen and STFC

Task 15.4: RF Superconductivity evaluation with QPR

Samp							
B-1	INFN SUBU	BUSTED! film	**REWELDING?**				
B-2	INFN SUBU	UNIVERSITÄT SIEGEN 2 µm Nb film	HZB RF test	CERN Recoat.: 7.3 µm Nb	CERN RF test	HZB RF test	multil./ new m.
B-3	INFN EP	UNIVERSITÄT SIEGEN 3 µm Nb film	HZB RF test	SIS: 3 µm Nb/30 nm AlN/100 nm NbN UNIVERSITÄT SIEGEN	HZB RF test		
B-4	INFN EP	3 µm Nb film	HZB RF test	INFN **EP**	**SIS**	HZB **RF test**	
B-5	INFN SUBU	3 µm Nb film	HZB RF test	RTU Laser treatment	HZB RF test	multil./new mat.	

DONE

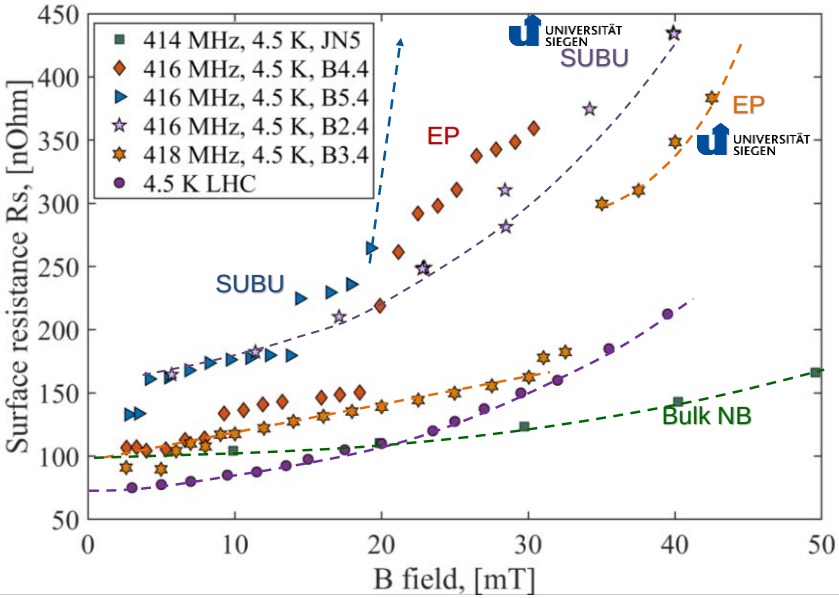
IN PROCESS

FAILED

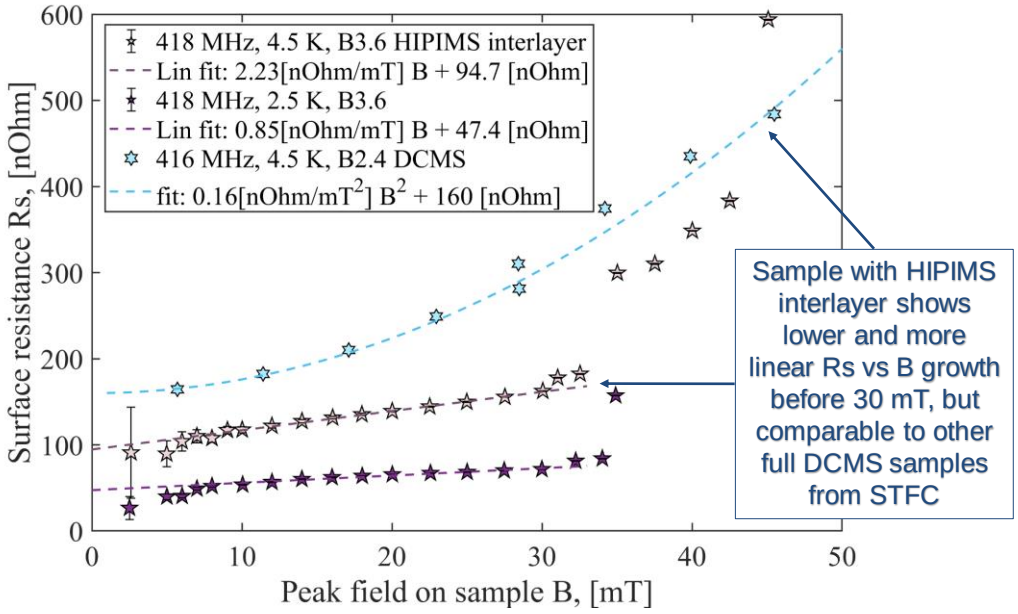
Courtesy of D. Tikhonov (HZB)

QPR testing at HZB

$R_s(B)$ at 415 MHz and T = 4.5 K



$R_s(B)$ at 415 MHz and T = 4.5 K and 2.5 K



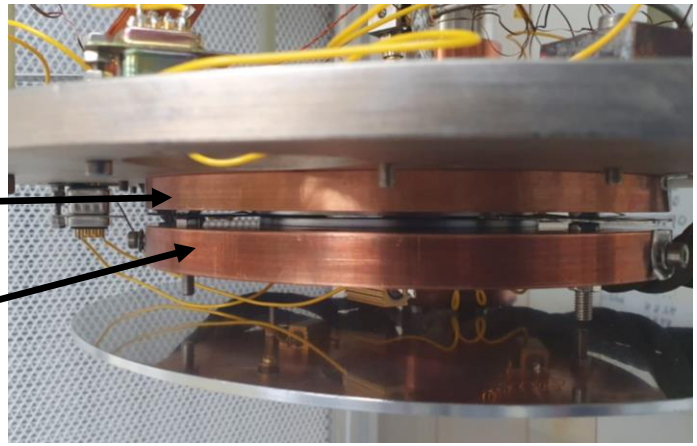
Q-switch problem

- Burrs on the edge may be a reason of "bad" film in those regions, which can overheat at some field values.
- IN future, the edges to be inspected and polished before coatings

Courtesy of D. Tikhonov (HZB)

Task 15.4: RF Superconductivity evaluation (STFC)

- At **ASTeC** a radiofrequency (RF) cavity and cryostat dedicated to the measurement of superconducting coatings at 7.8 GHz has been updated to operation with a closed-cycle refrigerator.
- Low power measurements with an emphasis on fast turn-around time.
- A cooldown demonstrated
  - T_{cavity} = 4.1 K and T_{sample} = 4.8 K.
- RF testing in progress



Pill-box cavity in a new facility with a closed-cycle refrigerator in STFC

Conclusions

- WP15 team works according to agreed plan
  - Development of superconducting films continued on small samples:
    - NbN, Nb_3Sn, NbTiN films as well as SIS structures have been deposited and tested
    - Small sample evaluation on SC properties is ongoing
    - Results reported
  - Evaluation of Nb films at RF conditions started
    - New QPR design
    - QRR sample polishing at INFN developed and applied to the samples
    - 5 QPR samples has been deposited at Siegen and STFC
    - QPR facilities at CERN and HZB are testing the samples produced by WP15 team
- Disruption due to COVID-19
- All activities will resume to normal as soon as the labs are open